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A COMPARISON OF VOID MEASUREMENT METHODS FOR CARBON/EPOXY COMPOSITES

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COMPOSITES DEVELOPMENT BRANCH

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ABSTRACT

This report studies four destructive measurement techniques for determining void volume fraction in CFRP composites. Two approaches to void measurement were taken: density determination/matrix digestion (DD/MD), and optical image analysis. Within each approach two techniques were studied. In the DD/MD approach, the water buoyancy technique (WBT) (see ASTM D 792) and density gradient technique (DGT) (see ASTM D 1505) were investigated. In the image analysis approach a Dapple Image Analyzer, and the more automated Omnimet Image Analyzer, techniques were investigated. It was found that "true" or absolute void content is quite difficult to measure regardless of the technique used. However, when making relative measurements between like specimens void content comparisons are reliable and practical to obtain. The WBT recorded consistently lower void content data than the DGT; it was also found to be less precise. For routine CFRP, void content determination, where relative comparisons are sufficient and high precision is not an issue, the WBT is recommended as it is practical to implement. When high precision is needed, the DGT is recommended. Image analysis methods produce highly localized data, but it is likely that they approximate true void content more closely than the DD/MD method because the void measurement, though actually a measure of void area, is direct. For more critical void content measurement where accuracy, as well as precision are required, a highly automated version of an image analysis technique, like the Omnimet, which scans a large number of cross sections is recommended. At present, this appears to be the best procedure available to determine true void content. DD/MD and image analysis approaches to void content determination are complimentary methods; the shortcomings of each are strengths of the other. Combining information from both methods is a superior means of characterizing voids in CFRP composites.

TERMINOLOGY

Throughout this report the following terms are limited to the strict definitions given below:

- Accuracy: The closeness of a measurement to the absolute or true value of that measurement.
- Precision: The relative reproducability of a measurement.
- Void content: A term interchangeable with void volume fraction and the symbol v_v.

NOMENCLATURE

W_l = weight of the laminate
W_f = fiber weight
W_m = matrix weight
ρ_e = experimentally measured laminate density
ρ_t = theoretical density of the laminate; i.e., density assuming no voids are present
ρ_f = manufacturer quality assurance data for carbon fiber density
ρ_m = manufacturer quality assurance data for matrix density
ρ_H = density of the heavy liquid
ρ_L = density of the light liquid
K_f = correction factor for acid reaction with carbon fiber
h_H = head height of the heavy liquid
h_L = head height of the light liquid
v_v = void volume fraction
σ = σ_{n-1} standard deviation
C_{ρ_e} = error of propagation coefficient for experimental density
C_{W_l} = error propagation coefficient for experimental laminate weight
C_{W_f} = error propagation coefficient for experimental fiber weight
C_{ρ_f} = error propagation coefficient for fiber density value
C_{ρ_m} = error propagation coefficient for matrix density value

CONTENTS

INTRODUCTION

Today, vacuum bag and autoclave cure methods for fabricating reproducible quality polymer composites are well established and standardized. As this field of composites processing continues to mature, the thrust of developmental work naturally shifts to areas of need. One current thrust area is an expanding effort to develop processing methods which decrease production costs without sacrificing part quality.

Clear relationships between void content and composite properties have been established.[1,2] In general, voids are an undesirable material defect whose presence in the matrix is particularly detrimental to interlaminar shear strength and fatigue life.

The degree of concern over void content in a composite part depends largely upon application. In many composite applications void content is quite critical, such as in advanced dynamic aerospace structures like helicopters where void contents above 1.5% are not tolerable. In this circumstance the autoclave process is invariably used. Vacuum bag processing is widely used to fabricate composite end items which are not critically dependent upon low void volume or high fiber volume fractions. Void content levels as high as 6% are often acceptable. There are many applications of this sort; e.g., ground vehicle components, secondary structural members, and more rudimentary composite end items.

In this report the subject matter is confined solely to a comparison of several destructive void measurement techniques. Carrying out this program properly entailed a detailed analysis of the methods of measuring voids in CFRP composites.

Two general types of destructive measurement methods were used: density determination/matrix digestion (DD/MD), and optical image analysis (IA). These two methods each applied two different techniques. The DD/MD method included both the water buoyancy technique (WBT) (see ASTM D792) and the density gradient technique (DGT) (see ASTM D1505). The IA method included two different optical image analysis systems: the Dapple IA system,*† and the Omnimet IA system.‡

These four techniques were used in a parallel study on the same two sets of CFRP specimens (one DD/MD set of 168, and one IA set of 60). The precision and accuracy of the void content data, as well as the differing trends inherent to each measurement technique, are presented.

*Fiber and matrix volume fractions are often attainable in this manner depending upon the contrast discrimination of the materials being analyzed.

†Dapple Image Analyzer, Dapple Sytems, Sunnyvale, CA.

‡Omnimet II Image Analyzer, Buehler, Lake Bluff, IL.

1 YOKOTA, M. J. *In Process Controlled Curing of Resin Matrix Composites.* SAMPE Journal, v. 14, no. 4, 1978, p. 11.

2 JUDD, N. C. W., and WRIGHT, W. W. *Voids and Their Effects on the Mechanical Properties of Composites - An Appraisal* SAMPE Journal, v. 14, no. 1, 1978, p. 10.

EXPERIMENTAL

Bulk Composite Void Measurement

Two commercially available CFRP epoxy prepreg tapes were used in this study to fabricate 30 laminates. They were Hercules AS4/3501-6 and Narmco T300/5208. The four void measurement techniques were applied to each of the 30 test groups. Each DD/MD technique was applied to the same 30 specimen sets (168 specimens total); each IA technique was applied to a separate set of 60 specimens cut from the same 30 laminates.

Density Determination

Two separate density determination methods were used on each CFRP specimen prior to matrix digestion. These were the WBT (see ASTM D792) and the DGT (see ASTM D1505, Method C).

Water Buoyancy Technique: The density of each composite specimen was measured according to the ASTM Standard. Through the known density of water, this technique gives a measurement of specimen density by two weighings: the specimen dry weight, then the buoyed weight of the same specimen suspended by a wire in a beaker of degassed distilled water. A laboratory balance with an accuracy of ± 0.001 g was used for all dry and wet weighings. The water density was corrected for temperature.

Density Gradient Technique: The DGT measures specimen density directly; no calculations are used. This technique is more involved than the buoyancy technique. Aqueous salt "heavy liquid" was chosen as the medium for density measurement. The density of the liquid medium is set by the amount of a complex salt dissolved into water. Liquid densities ranging from 1.0 g/cc to 2.5 g/cc could be made with these solutions. The apparatus, as shown in Figure 1, includes a 70 cm glass column marked with antiparallax grids for accurate height measurement (± 0.5 mm), a Plexiglas water jacket, and a circulating temperature control unit which kept the water in the jacket closely controlled at 23°C ± 0.5°C. This apparatus kept the fluid gradient convectively stagnant and stable. A peristaltic pump fitted with a three-roller pump head sized for No. 14 Tygon tubing was used to fill the column. This pumping setup filled the column slowly and at a constant linear rate. The density of the solution drawn from flask A increased linearly with time. As the lower density or "light" liquid is pumped in the column from flask A, it is replaced by the "heavy" liquid which enters flask A from flask B. Flow of the heavy liquid is driven by the slight head pressure created by the steady removal of light liquid from flask A. Slow filling is important to establishing a linear gradient and also helps keep the gradient convectively stagnant and stable. It took about seven hours to complete each column fill. This procedure was reliable and resulted in stable, linear columns.

Once filled in this manner, an undisturbed column remained linear and ready for use for an extended period of time ranging over several weeks, although the top five centimeter region of the column would predictably destabilize within 24 hours. It is not known why this occurred. Away from this region the gradient was stable and the lower depths of the column were used for density measurement.

Five calibrated sink floats (± 0.0005 g/cc) were wetted in a separate beaker of the light liquid solution and slowly immersed into the filled column with tweezers. Next, approximately 10 CFRP specimens were wetted in the light solution and slowly immersed into the column. The floats and specimens were then left to settle for two hours. The column calibration line was determined by finding the least squares linear fit of the float standard density value versus height. Correlation coefficients were 0.995 or higher for all columns. Turbulence created by immersing the 15 objects into the column had a remarkably slight effect on gradient linearity and stability in spite of the fact that the CFRP specimens were 2.5 cm x 2.5 cm x 0.3 cm in size.

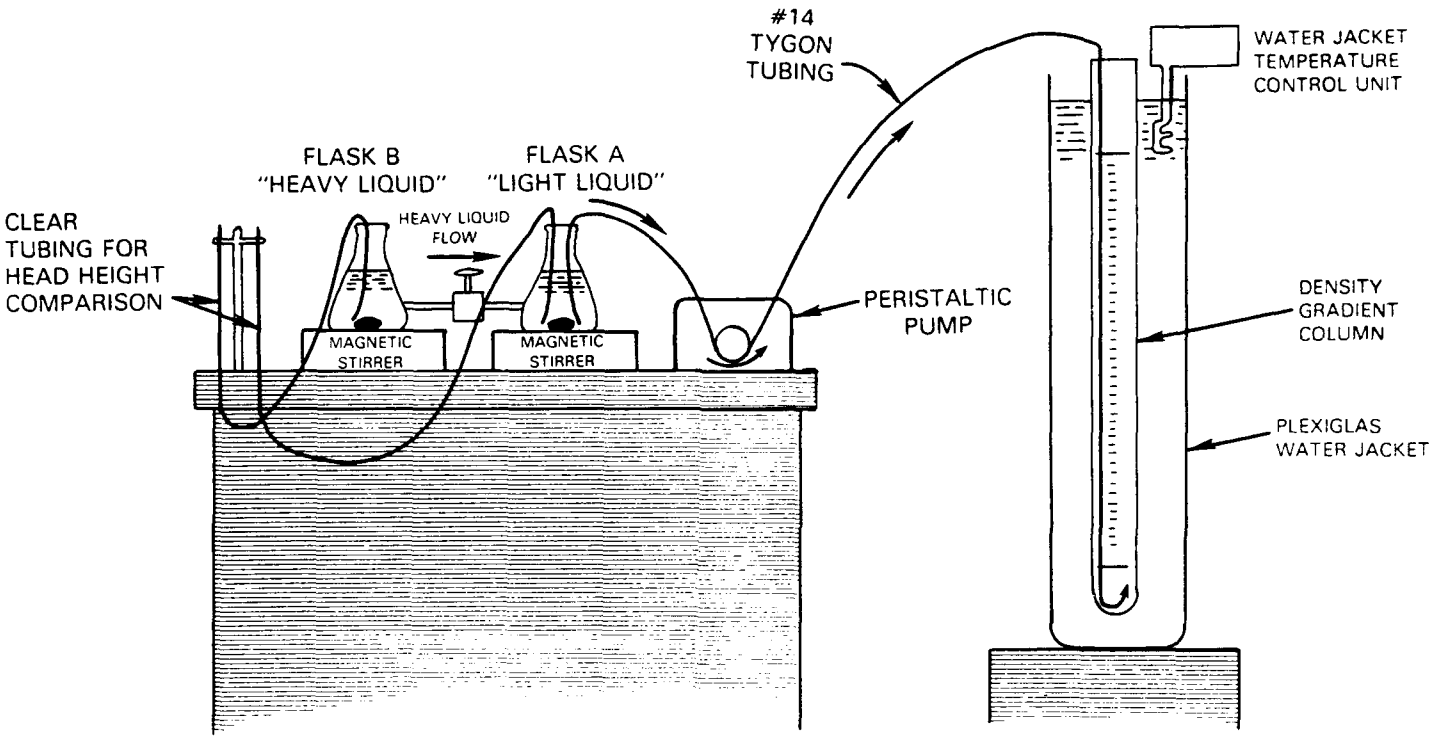

Figure 1. Apparatus for measuring density by the density gradient technique.

The CFRP specimens were rectangular and the center of gravity was assumed to coincide with the center of geometry. Height measurement of the center of geometry was made by locating the height of the top and bottom most edges of the specimen; the height of the center of geometry is midway between these points.

Controlling the gradient range and sensitivity was an inexact procedure. However, empirically learned relations served well as a guide to getting actual column values close to the target values (see Appendix A). Each column pumped was slightly different than the next but all were close to 0.010 g/cc/cm in sensitivity. Column sensitivity was controlled by the density difference between light and heavy liquid starting densities. The column low density value was controlled by the equilibrated density of the starting light liquid.

Matrix Digestion

Once the two density tests were complete the specimen fiber weight was determined by matrix digestion (see ASTM D3171). The two epoxies used in this study were both suited to digestion in heated concentrated nitric acid. CFRP specimens weighing approximately two grams were placed in a 600 cc beaker with 75 cc of concentrated nitric acid. The beaker was covered (but not sealed) and placed in a ventilated hood on a hotplate and heated to 80°C. After two hours the beaker was removed and the bare fibers rinsed with water. They were washed two rinses beyond the point where no sign of the reaction residue was visible to the eye. This was typically five rinses. The fibers were then thoroughly dried at 115°C for six hours and left to cool overnight. The next day the fiber weight was determined on the laboratory balance.

Control tests were run on the T300 and AS4 fibers to determine the correction factor, K_f, for acid reactions with the carbon fibers. In the control tests bare carbon fibers were weighed and run through the same matrix digestion procedure as the composite specimens. After drying they were weighed again. The ratio of the final weight to the starting weight is the correction factor K_f.

Once specimen density and fiber weights were known, specimen theoretical density and void volume fraction were calculated by the following relations:

$$v_v = 1 - \rho_e / \rho_t \tag{1}$$

$$\rho_t = W_\ell / [(K_f W_f / \rho_f) + (W_m / \rho_m)] \tag{2}$$

Void volume fraction was calculated for each specimen using both the water buoyancy-determined and gradient column-determined experimental density data sets. The manufacturer quality assurance specifications were used in these calculations for values of fiber and matrix density.

Optical Void Measurement

Two IA systems were used to determine void content in the CFRP laminates. These systems were the Dapple IA system, and the Omnimet IA system. Both methods allowed quantitative, as well as qualitative, inspection of the CFRP specimens.

Specimen Preparation

Both IA methods used the same set of 60 specimens for study. Approximately square specimens (2.5 cm x 2.5 cm) were cut from the central area of the test laminates for materiallographic preparation. These specimens were encapsulated in mounting epoxy for polishing (see Figure 2). They were cut in half at 45° to the 0/90 fiber direction (see Figure 3). This cross-sectional angle eliminated fiber pullout during polishing. Also, the 45° orientation produced uniform optical contrast of the fibers for optimal IA measurement. A 0/90 cross-sectional view specimen was also mounted, polished, and viewed for qualitative assessment of void location through the laminate thickness.

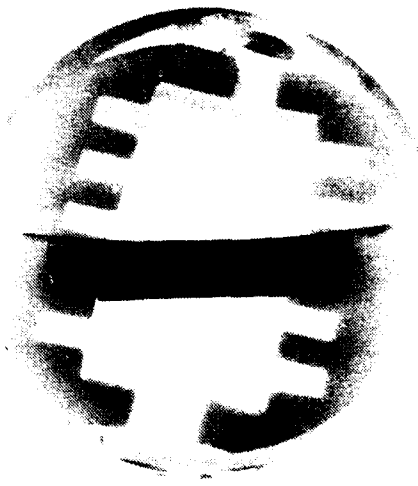

Figure 2. A CFRP specimen mounted and ready for quantitative optical image analysis.

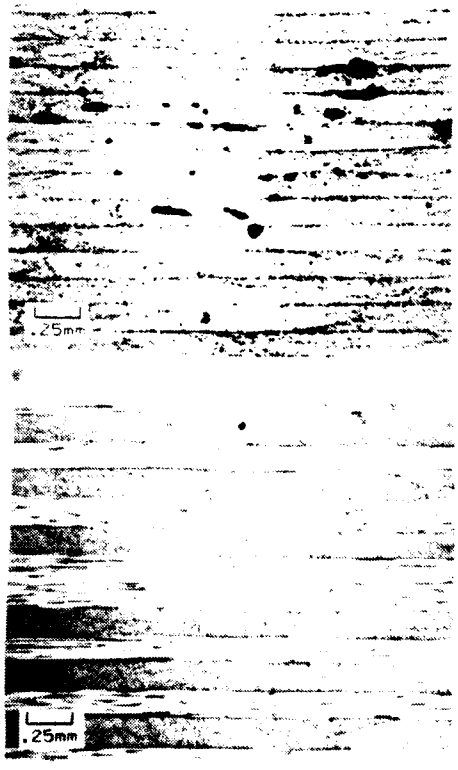

Figure 3. Above, cross-sectional view of a $(0/90)_{4s}$ laminate seen at 45° to the fiber direction. Black areas are voids. Below, a $(0/90)_{4s}$ laminate seen at 0° and 90° to the fiber directions.

Dapple Image Analysis

The Dapple IA system was used to measure the average void area fraction (which is equivalent to void volume fraction) of each CFRP cross section.[3]

In the Dapple technique the entire area of each polished cross section was first viewed qualitatively with a light microscope to observe the overall laminate. Four representative areas of the cross section surfaces were then chosen and photographed at 40X magnification. The actual sampling area was 4.5 mm^2 to 5.0 mm^2 per photograph. The total sampling area per specimen was 18 mm^2 to 20 $mm.^2$ The photographs were then measured for void area fraction using the Dapple IA system, which calculated the average void area fraction over the entire photographic image. The Dapple IA system measures features of area acquired from video images. The hardware consists of a personal computer, TV camera, 9" TV monitor (for actual image observation), 12" monochrome high resolution monitor (for image processing and measurement), and conventional lighting apparatus. The accompanying software package digitizes and measures the video images. For each video image the brightness is measured in 64 gray level steps at each point in a 254 x 192 pixel array. The software allows the operator to separate the areas or features of interest from the background via gray level discrimination. These detected or discriminated areas can then be stored, reprocessed, measured, and statistically analyzed.

Omnimet Image Analysis

The Omnimet also discriminates and measures video images. However, this personal computer-based system is interfaced directly to an optical microscope via a Vidicon high resolution camera. This system offers increased magnification and allows for direct measurement of voids from polished specimens. The polished CFRP laminate cross sections were imaged and measured at 250X magnification via the optical microscope. The voids were discriminated and measured for each area viewed. Approximately 2 mm^2 of specimen area was measured for each specimen. This is an area 10 times smaller than that measured by the Dapple technique. The areas were measured through the entire thickness at 60 random points over the cross section. In addition to void area fraction, the Omnimet system can be used to measure fiber and matrix area fraction.

RESULTS AND DISCUSSION

An in-depth look at what is involved in generating the void data in the four techniques was studied and quantitative exposure of the strong and weak areas of each method was accomplished.

Thirty void data sets; i.e., 30 distinct CFRP cured laminates, were generated for each of the four void measurement techniques. A total of 168 DD/MD specimens and 60 optical IA specimens were each measured by two techniques for a grand total of 456 void measurements. This large amount of data provided a solid compilation which enabled trends in void content determination to be uncovered.

3. UNDERWOOD, E. E. *Quantitative Stereology*. Addison-Wesley, Massachusetts, 1970. p. 27.

DD/MD Results

Table 1 lists the density data sets generated for the WBT and DGT, respectively, along with the theoretical density for each data set. Since there is no connection between data sets, e.g., each laminate was processed in a separate run and under several different processing conditions, the values for average density and average error are not related to each other. However, each value is separately significant and useful.

The data in Table 1 reveals several clear trends of the two DD/MD methods. The WBT consistently recorded a slightly higher density value than that of the DGT. Of the 30 data sets, 21 recorded a higher density value when measured by the WBT. It is unclear which technique reflects the true density more closely.

Measurement precision was higher for the DGT. For the 30 data sets an average error of ± 0.009 g/cc was measured for the WBT, while the DGT average error was ± 0.006 g/cc. Twenty-three of the 30 data sets were recorded as more precise when measured by the DGT.

These density values were put into the same computation for v_v (see Equation 1). The resulting data, shown in Table 2, is a reflection of the same trends just discussed. The WBT recorded a lower average v_v (1.1% compared to 1.4%) and the average value was less precise (± 0.52% compared to ± 0.36%). Both methods recorded some negative values for v_v. Since the WBT recorded a lower average v_v, more negative values would be expected, and this was indeed the case. The WBT recorded 10 of the 30 data sets as having a v_v less than zero, while the DGT recorded six. This does not, however, lead to a conclusion that the WBT void volume fraction values are less accurate. The density measurement is not the only value in the computation which is not known with certainty to be accurate. Aside from the laminate weight measurement, which is felt to be very accurate (as well as precise), the remaining three parameters are not known to be accurate with any certainty. Since this testing was unable to isolate the accuracy of four of the five parameters, it cannot be determined which density measurement technique more closely matches the true density of the laminate. It can, however, be said that the DGT is more precise and, therefore, gives better results when making relative comparisons. This improved precision comes as a result of higher skill, special equipment, and a larger investment in the testing time of the DGT. The choice of whether to use the DGT is made on the basis of whether these tradeoffs are worth the gain in measurement precision.

The two DD/MD tests share the same five sources of experimental error. These are:

- Experimental laminate density measurement
- Laminate weight measurement
- Fiber weight measurement
- Fiber density from manufacturer supplied specifications
- Matrix density from manufacturer supplied specifications

Table 1. DENSITY DATA GENERATED BY THE WATER BUOYANCY TECHNIQUE AND THE DENSITY GRADIENT TECHNIQUE ALONG WITH THE THEORETICAL DENSITY FOR EACH OF THE 30 DATA SETS.

			Mean Densities		Standard Deviation	
Data Set	Specimen Count	Mean Theo. Density (g/cc)	WBT (g/cc)	DGT (g/cc)	WBT (g/cc)	DGT (g/cc)
1	6	1.564	1.570	1.569	0.022	0.012
2	6	1.552	1.563	1.560	.025	.009
3	5	1.566	1.574	1.564	.008	.008
4	5	1.600	1.615	1.610	.011	.003
5	6	1.613	1.631	1.626	.016	.002
6	6	1.586	1.592	1.585	.006	.004
7	6	1.595	1.617	1.569	.010	.004
8	5	1.615	1.597	1.570	.020	.008
9	6	1.587	1.548	1.560	.017	.006
10	6	1.574	1.544	1.545	.009	.004
11	4	1.591	1.547	1.548	.010	.008
12	6	1.591	1.539	1.538	.009	.005
13	6	1.600	1.561	1.562	.008	.007
14	5	1.599	1.547	1.541	.008	.006
15	7	1.571	1.556	1.555	.005	.008
16	7	1.580	1.560	1.564	.007	.003
17	7	1.585	1.566	1.569	.007	.004
18	7	1.584	1.546	1.557	.013	.006
19	6	1.586	1.538	1.531	.004	.006
20	6	1.551	1.566	1.558	.010	.015
21	4	1.533	1.533	1.514	.005	.007
22	5	1.562	1.527	1.528	.004	.006
23	5	1.558	1.530	1.531	.006	.005
24	6	1.555	1.516	1.512	.005	.008
25	5	1.555	1.510	1.507	.012	.005
26	6	1.554	1.507	1.501	.002	.004
27	6	1.586	1.580	1.557	.002	.011
28	5	1.567	1.580	1.581	.006	.002
29	4	1.579	1.576	1.576	.003	.003
30	4	1.605	1.587	1.583	.006	.005
Average		1.578	1.561	1.556	0.009	0.006

Table 2. VOID VOLUME FRACTION DATA GENERATED BY THE WATER BUOYANCY TECHNIQUE AND THE DENSITY GRADIENT TECHNIQUE OF THE 30 DATA SETS.

Data Set	Specimen Count	Mean Void Volume Fraction		Standard Deviation	
		WBT (%)	DGT (%)	WBT (%)	DGT (%)
1	6	-0.43	-0.35	0.52	0.31
2	6	-0.73	-0.49	1.53	0.09
3	5	-0.52	0.16	0.15	0.08
4	5	-0.97	-0.64	0.62	0.04
5	6	-1.11	-0.78	0.82	0.28
6	6	-0.39	0.06	0.35	0.46
7	6	-1.35	1.63	1.02	0.89
8	5	1.12	2.78	0.96	0.36
9	6	2.45	1.70	0.48	0.29
10	6	1.86	1.82	0.91	0.17
11	4	2.76	2.73	0.33	0.13
12	6	3.23	3.29	0.65	0.58
13	6	2.40	2.35	0.30	0.27
14	5	3.30	3.66	0.29	0.25
15	7	0.95	1.02	0.26	0.26
16	7	1.25	1.01	0.37	0.13
17	7	1.22	1.01	0.27	0.19
18	7	2.40	1.73	0.20	0.35
19	6	3.00	3.43	0.63	0.54
20	6	-0.94	-0.41	0.26	0.39
21	4	-0.02	1.23	0.40	0.59
22	5	2.23	2.17	0.44	0.29
23	5	1.80	1.76	0.33	0.33
24	6	2.46	2.75	0.24	0.38
25	5	3.04	3.06	0.34	0.36
26	6	3.00	3.40	0.24	0.24
27	6	0.43	1.86	0.78	0.23
28	5	-0.85	-0.90	0.17	1.41
29	4	0.15	0.15	1.47	0.21
30	4	1.12	1.32	0.26	0.67
Average		1.10	1.42	0.52	0.36

Statistical backtracking to get a better feel for the significance of the various error sources was also undertaken in this study. All error sources were assumed to be independent of one another. This was felt to be a valid and intuitively obvious assumption. Accepting this premise, the propagation of measurement errors through equations were able to be traced and their contribution to the final error in v_v estimated. This is derived in Appendix B.

The error contribution to v_v from every source was not able to be isolated. However, by lumping the first, third, fourth, and fifth above values the error contribution from the laminate weight measurement was isolated. When measured data for the above five listed parameters are combined with the expanded form of Equation (B2) which is Equation (B5) in Appendix B, the contribution of error from the laminate weight measurement can be estimated.

$$\sigma_{v_v} = [(C_{\rho_c}\ \sigma_{\rho_c}) + (C_{w_l}\ \sigma_{w_l})^2 + (C_{w_f}\ \sigma_{w_f})^2 + (C_{\rho_f}\ \sigma_{\rho_f})^2 + (C_{\rho_m}\ \sigma_{\rho_m})^2]^{1/2} \qquad \text{(B5)}$$

Equation B5 is generic to all DD/MD methods; it applies to any material and any method of composite density and fiber weight determination for tracing the error in void volume fraction; e.g., fiber burnout of a glass reinforced composite.

This analysis brings out some notable points. First, the error of the measurement alone does not determine the effective error. It is altered by its coefficient in the calculation. The coefficients are listed in Table 3 in order of decreasing size. It is seen that all five coefficients are less than one, thus they all serve to lessen the effect of their respective error terms. The laminate density measurement is by far the highest penalized value, followed by the fiber and matrix density values (approximately a factor of four times less penalized), and the fiber and laminate weight values (10 and 20 times less penalized, respectively). This analysis indicates that careful measurement of the laminate density is critical to obtaining meaningful void volume fraction data. The remaining parameters are not as sensitive to error propagation through the void volume fraction content calculation.

Table 3. SQUARED ERROR PROPAGATION COEFFICIENTS FOR THE FIVE ERROR SOURCES.

Error Source	WBT Coeffs. Squared	DGT Coeffs. Squared	Ratio Factor
Laminate Density $(g/cc)^2$	0.40160	0.40160	20.2
Fiber Density $(g/cc)^2$	0.11610	0.11586	5.8
Matrix Density $(g/cc)^2$	0.09618	0.09556	4.8
Fiber Weight $(g)^2$	0.03924	0.03899	2.0
Laminate Weight $(g)^2$	0.01991	0.01978	1.0

The error in the laminate weight is inconsequential when compared to the magnitude of error introduced by the density measurement and the remaining three error sources. In addition, the error propagation coefficient for the laminate weight happens to be the smallest of

the five coefficients; the density measurement error coefficient is the largest by a wide margin. In fact, it is larger than the other four coefficients combined. It is suspected that the laminate density measurement and fiber weight measurement are the two major causes of lost precision. It is reasonable to assume that the manufacturers values for fiber and resin density, while perhaps not accurate, are quite reproducible from specimen to specimen. This cannot be said for the laminate density measurement and fiber weight measurement which are subject to experimental variation. The error in the laminate weight measurement ostensibly has no effect on the accuracy or precision of the final void volume fraction value.

IA Results

Another much different approach to measuring v_v in CFRP composites is optical IA. The differences between this approach and the DD/MD approach are discussed in detail in the Experimental Section of this report. Two specimens from each of the 30 CFRP laminates were cut, mounted, and polished for IA measurement. As with the DD/MD methods, two separate IA methods were used: the Dapple technique and the Omnimet technique. These results are shown in Table 4.

Unlike the DD/MD methods, IA methods involve no calculation of v_v. In the IA method the laminate interior cross section is exposed and void area fraction, which is equivalent to v_v, is measured directly by optical means. In addition to the v_v measurement, the size, shape, and location of voids can be clearly seen. With all this information to offer, IA methods would seem to be vastly superior to DD/MD methods. Unfortunately, IA has drawbacks of its own, the most severe of which is an inability to measure these features in bulk. An IA image is one thin cross-sectional view which cannot be safely assumed to represent the entire laminate. *It is an extremely localized measurement.*

In Table 4 it is seen that neither IA method recorded a negative value for v_v; in fact, IA will never record a negative v_v because the laminate interior is being viewed directly. Knowing this, it is easy to trust an IA measurement value as being close to the true void content (for that location in the laminate). Although it is likely that carefully obtained IA results do approximate the true v_v better than the DD/MD method, there are considerations to be wary of here as well. Subtle problems can occur which only a skilled experimenter would realize. In the specimen mounting step the potting resin can fill in voids, masking their existence. In the polishing step the cross section can be scratched, or worse chipped, to form a false void not easily detected without the experience to visually identify a chip from a naturally occurring void. Scratches are more easily identified as they typically appear as finite straight "void lines" in the optical image. Lastly, in the measurement step differences in optical contrast can shift resulting data values.

Two results are particularly significant in Table 4. The recorded v_v is clearly higher when measured by an IA technique. This result confirms previous results found at this laboratory.[4] Sixty specimens were measured in each case so the average v_v value represents a large number of cross sections. The average error from the 30 laminates was $\pm$ 0.20% for the Omnimet technique. The other three methods, including the Dapple technique, were less precise than the Omnimet technique.

4. RICCA, J. J., JURTA, R. M., AND DADY, C. Unpublished results, 1986.

Table 4. VOID VOLUME FRACTION DATA OF THE 30 DATA SETS GENERATED BY THE DAPPLE AND OMNIMET TECHNIQUES

Data Set	Specimen Count	Mean Void Volume Fraction		Standard Deviation	
		Dapple (%)	Omnimet. (%)	Dapple (%)	Omnimet. (%)
1	2	0.00	0.00	0.00	0.00
2	2	0.00	0.00	0.00	0.00
3	2	3.30	4.50	0.40	0.15
4	2	0.00	0.00	0.00	0.00
5	2	0.00	0.00	0.00	0.00
6	2	1.91	2.00	0.31	0.21
7	2	2.73	2.30	1.68	0.26
8	2	4.10	4.00	1.30	0.00
9	2	1.70	1.80	0.40	0.26
10	2	2.00	1.90	0.00	0.28
11	2	2.70	2.10	0.40	0.35
12	2	2.20	2.00	0.30	0.07
13	2	4.50	2.40	0.10	0.28
14	2	5.60	3.70	3.50	0.28
15	2	1.00	0.90	1.60	0.11
16	2	1.20	0.70	0.80	0.56
17	2	0.70	0.10	0.40	0.00
18	2	2.10	1.60	0.80	0.49
19	2	1.70	2.00	1.00	0.28
20	2	2.09	2.00	1.33	0.25
21	2	4.45	4.30	1.61	0.24
22	2	5.80	5.20	1.30	0.14
23	2	2.40	1.90	0.50	0.35
24	2	3.60	3.20	0.80	0.16
25	2	6.40	4.90	0.80	0.00
26	2	4.40	4.80	0.60	1.34
27	2	0.00	0.00	0.00	0.00
28	2	0.00	0.00	0.00	0.00
29	2	0.00	0.00	0.00	0.00
30	2	0.00	0.00	0.00	0.00
Average		2.22	1.94	0.66	0.20

A fairly wide discrepancy resulted in the precision of the two optical methods. The overall error was ± 0.66% for the Dapple technique and ± 0.20% for the Omnimet technique. This result was expected as the Omnimet technique is automated and scans 60 random cross-sectional views. The Dapple technique samples just four areas of the cross section. Examining the results, only data set No. 26 showed an error greater than 1% when the Omnimet technique was used. The Dapple technique, on the other hand, recorded eight incidents of error greater than 1%.

Intertechnique Trends

When the four methods are compared (see Table 5) other trends become evident. It has been shown that, in relative comparisons, all four methods are in rough agreement when measuring v_v. It has also been shown that the DGT and the Omnimet technique gave results which were more precise, and the tradeoffs for this in both cases were a need for higher skill level, special, more expensive equipment, and an increased investment in time.

Void content can be a difficult quantity to measure with confidence, but much less so when measuring in a relative sense. It is quite difficult to determine true void content. Past work on CFRP composites at this laboratory has shown that fiber content determined using DD/MD methods correlate closely with quantitative IA data.[4] However, values for void content have been found to vary between these methods. Void content determined from DD/MD methods are typically lower (even negative) compared to IA results due to errors introduced and propagated in the void content calculation.[4]

DD/MD methods provide an overall average relative value of void content and can be rerun easily to obtain a solid data base. However, this approach gives no insight as to the size, shape, or location of voids. IA methods, on the other hand, provide direct evidence of the existence, relative size, and location of voids within the laminate. However, IA methods rely on an extremely localized sampling area, and one or two cross-sectional views do not constitute a large enough data base to draw decisive conclusions about the void content of the laminate as a whole. In order to draw conclusions about the laminate as a whole from a few cross-sectional views, one must assume and rely upon random distributions of voids and fiber throughout the laminate. This is an unwise assumption. Many repetitions would get around this problem, but this is not practical as the specimen preparation and measurement process is involved and time consuming. Also, optical contrast standards do not yet exist for CFRP composite materials so the true accuracy cannot be determined. However, the measurement reproducibility for a single specimen is within ± 2% for CFRP materials, so the data can be used to compare relative differences.[4]

Table 5. COMPARISON OF DD/MD VERSUS IA AVERAGE VOID VOLUME FRACTIONS.

Data Set	Mean Void Volume Fraction		Standard Deviation	
	DD/MD (%)	IA (%)	DD/MD (%)	IA (%)
1	-0.39	0.00	0.42	0.00
2	-0.61	0.00	0.81	0.00
3	-0.18	3.90	0.12	0.28
4	-0.80	0.00	0.33	0.00
5	-0.94	0.00	0.55	0.00
6	-0.16	1.95	0.41	0.26
7	0.14	2.50	0.95	0.97
8	1.95	4.05	0.66	0.65
9	2.07	1.75	0.38	0.33
10	1.84	1.95	0.54	0.14
11	2.74	2.40	0.23	0.38
12	3.26	2.10	0.62	0.19
13	2.38	3.45	0.29	0.19
14	3.48	4.65	0.27	1.89
15	0.98	0.95	0.26	0.86
16	1.13	0.95	0.25	0.68
17	1.12	0.40	0.23	0.20
18	2.07	1.85	0.28	0.65
19	3.22	1.85	0.58	0.64
20	-0.67	2.05	0.33	0.79
21	0.60	4.40	0.50	0.93
22	2.20	5.50	0.37	0.72
23	1.78	2.15	0.33	0.43
24	2.60	3.40	0.31	0.48
25	3.05	5.65	0.35	0.40
26	3.20	4.60	0.24	0.97
27	1.14	0.00	0.50	0.00
28	-0.87	0.00	0.79	0.00
29	0.15	0.00	0.84	0.00
30	1.22	0.00	0.47	0.00
Average	1.26	2.08	0.44	0.43

Difficulty arises with all methods when the question of accuracy is addressed. The DD/MD methods consistently recorded much lower v_v (an average of 1.2%) than specimens cut from the same 30 plates measured by optical means (an average of 2.1%). This illustrates just how difficult it is to determine true void content. Thirty CFRP laminates were measured by two accepted methods of determining v_v and, despite a large number of measurements within each technique, it was found that they not only disagree, but disagree by a significant margin; virtually one full percent. When considering v_v, especially below the 4% range, this is a large discrepancy. The question then arises as to which set of results are more accurate and, further, how to verify this. This study indicates that the accuracy of v_v cannot be verified in any practical way. To know the true v_v with certainty is only possible with extremely high skill level on the part of the experimenter who has a large empirical data base on which to rely. On the other hand, determining v_v for a relative measurement for comparisons between specimens is fairly easily done. A measurement precision in the range of ± 0.5% can be obtained with a modest time investment in learning any of these various test techniques. This is proven in Table 5 where, after all void content measurement errors are averaged, a common value of about ± 0.4% resulted in both methods.

SUMMARY

The WBT recorded consistently higher density values than the DGT, which translates to consistently lower void content data. It is not clear which technique reflects the true density and void content more closely. Measurement precision is higher for the DGT.

For routine CFRP void content determination, where relative comparisons between test groups is sufficient, the WBT should be used. Careful attention to procedure will result in measurement precision near ± 0.5%. The DGT is capable of substantially improving the precision of the density measurement, easily doubling it, but this gain will be lost in the error of the other measurements involved in the void content calculation. Experimental errors for WBT can be traced, isolated, and estimated by the statistical procedure given in this report.

For more critical void content measurement where accuracy, as well as precision, is required this study indicates that a highly automated version of an IA technique, like the Omnimet, be used to view a large number of cross sections to get around the problem of localized measurement. At the present time this appears to be the best procedure available to measure true void volume fraction.

The good relative measurement agreement between DD/MD and optical IA methods for determining void volume fraction in CFRP laminates indicates that either void characterization method can stand on its own as long as the precision of a particular technique is acceptable for test application. IA methods are localized but provide more information. They are likely to represent void content more accurately when done with care due to the fact that the measurement, though a two-dimensional measurement of void area fraction, is direct.

DD/MD and cross-sectional optical IA methods are complimentary techniques; as such, the degree of consistency between the methods provides a measure of confidence in the data.

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APPENDIX A: CONTROL OF DENSITY GRADIENT COLUMN RANGE AND SENSITIVITY

Mix 1000 cc light, 1000 cc heavy, and 200 cc light in a third beaker for specimen prewetting.

Fill the column to about 5 cm above the top height marker on the column; simply calculating $\pi r^2 \ell$, this is approximately 95 cc of solution. To fill the column grid zone takes $\pi r^2 \ell$, which for this column was 1295 cc. The bottom of the column beneath the grid zone is also filled but this area is not cylindrical. To obtain this volume accurately, water was poured out of a graduated cylinder until the zero mark was reached. The displaced volume was 70 cc for this column.

Summing these volumes gives 1460 cc. This is the amount of fluid which will actually enter the column. The remaining 540 cc is left in the flasks as excess.

Example of Light and Heavy Liquid Starting Density Calculation

The target goals are:

- Solution density at 70 cm column height = 1.47 g/cc
- Solution density at 0 cm column height = 1.54 g/cc

This works our to a column sensitivity of (0.07 g/cc)/70 cm or 0.010 g/cc/cm.

To reach these goals, mix the initial light liquid to a density of 0.005 g/cc lower than the 70 cm target value or 1.465 g/cc. Mix the initial heavy liquid to 0.03 g/cc higher than the desired 0 cm density or 1.57 g/cc. When the stop cock is opened and flasks A and B are connected they will equilibrate according to the following relation:

$$\rho_H h_H = \rho_L h_L \qquad \text{(A1)}$$

If the following values are known to be:

h_L = 14.0 cm

ρ_H = 1.57 g/cc

ρ_L = 1.47 g/cc

then, h_H can be approximated using Equation A1 to be 13.2 cm. In other words, if the head height of the heavy liquid is 13.2 cm above the outlet of flask B, there will be a minimal amount of equilibration taking place and the starting density in the column will have been controlled.

This defines the equilibrated head height difference between flasks A and B. Add the full 1000 cc of light liquid to flask A. This results in a head height of approximately 14.0 cm above the flask inlet. Using the above equation, the pre-equilibrated density head height of the heavy liquid needs to be 13.2 cm. When flask B is filled to this height the two solutions will be somewhere near equilibrium before the stop cock is opened. This procedure is not exact, and when the stop cock is opened some liquid will flow either forward or

backward between flasks until the head pressures equalize. The peristaltic pump should not be started until ample time for equilibration is given. Usually the liquid flow can be seen in the form of striation lines at the interface where the two solutions mix. When these disappear the fluids are equilibrated and the column filling process can begin.

A good way to compare head heights is to insert a clear Tygon tube into each flask, siphon them full of the respective liquids, then place the tubes side by side. The height of the liquid in the tube will match the height of the liquid in the flask and the difference between the two can be clearly monitored in this way.

APPENDIX B: TRACING ERROR PROPAGATION

The general relation for propagation of independent errors is given by

$$\sigma^2 = \sum_{i=1}^{N} (\partial f / \partial x_i)^2 \sigma_{x_i}^2 \tag{B1}$$

The relation for determining v_v in a DD/MD experiment is given by Equation 1. This relation looks simple, but actually is not. The value ρ_e is determined by experiment; this is not the case with ρ_t, which must be calculated. The relation for ρ_t is given by Equation 2. Combining Equations 1 and B1, the unexpanded relation for σ_{v_v} is

$$\sigma_{v_v} = [(1/\rho_t^2)\, \sigma_{\rho_e}^2 + (\rho_e/\rho_t^2)^2\, \sigma_{\rho_t}^2]^{1/2} \tag{B2}$$

Equations B2 arises by combining Equations 1 and B1. When Equation 2, the expression of ρ_t in basic parameters, is substituted a lengthy term-by-term expression for σ_{v_v} results. Repeating Equation 2, we have

$$\rho_t = W_\ell / [(K_f\, W_f/\rho_f) + (W_m/\rho_m)] \tag{2}$$

The error in ρ_t will result in four additive terms derived from the general expression

$$\sigma_{\rho_t}^2 = (\partial_{\rho_t}/\partial W_\ell)^2 \sigma_{w_\ell}^2 + K_f^2 (\partial \rho_t / \partial W_f)^2 \sigma_{w_f}^2 + (\partial \rho_t / \partial \rho_f)^2 \sigma_{\rho_f}^2 + (\partial \rho_t / \partial \rho_m)^2 \sigma_{\rho_m}^2 \tag{B3}$$

A step-by-step calculation will not be done here. Combining Equations 2 and B3 and doing the necessary calculation, five coefficients of error propagation are found. They are:

Laminate density coefficient: $1/\rho_t$

Laminate weight coefficient:
$(\rho_e/\rho_t^2)\,[(K_f W_f/\rho_f + W_m/\rho_m)^{-1} - W_\ell/\rho_m (K_f W_f/\rho_f + W_m/\rho_m)^{-2}]$

Fiber weight coefficient: $(\rho_e/\rho_t^2)\,[-W_\ell (1/\rho_f - 1/\rho_m)(K_f W_f/\rho_f + W_m/\rho_m)^{-2}]$

Fiber density coefficient: $(\rho_e/\rho_t^2)\,[W_\ell K_f W_f/\rho_f^2 (K_f W_f/\rho_f + W_m/\rho_m)]^{-2}$

Matrix density coefficient: $(\rho_e/\rho_t^2)\,[W_\ell W_m/\rho_m^2 (K_f W_f/\rho_f + W_m/\rho_m)]^{-2}$

The above five coefficients squared, then multiplied by the square of their respective error, and added together, make up Equation B4 (which will not be listed out in line form) and are equal to $\sigma_{\rho_t}^2$. Combining this expression with Equation B2, an expanded form of Equation B2 is

$$\sigma_{v_v} = [C^2_{\rho_e}\,\sigma^2_{\rho_e} + C^2_{w_\ell}\,\sigma^2_{w_\ell} + C^2_{w_f}\,\sigma^2_{w_f} + C^2_{\rho_f}\,\sigma^2_{\rho_f} + C^2_{\rho_m}\,\sigma^2_{\rho_m}]^{1/2} \qquad \text{(B5)}$$

The combination of Equations B4 and B5 are the fully expanded form of Equation B2.

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A COMPARISON OF VOID MEASUREMENT
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Seth R. Ghiorse

Technical Report MTL TR 91-13, April 1991, 24 pp-
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A COMPARISON OF VOID MEASUREMENT
METHODS FOR CARBON/EPOXY COMPOSITES
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This report studies four destructive measurement techniques for determining void volume fraction in Carbon/Epoxy composites. Two approaches to void measurement were taken: density determination/matrix digestion (DD/MD), and optical image analysis. Within each approach two techniques were studied. In the DD/MD approach, the water buoyancy technique (WBT) (see ASTM D 792) and density gradient technique (DGT) (see ASTM D 1505) were investigated. In the image analysis approach a Dapple Image Analyzer, and the more automated Omnimet Image Analyzer, techniques were investigated. It was found that "true" or absolute void content is quite difficult to measure regardless of the technique used. However, when making relative measurements between like specimens void content comparisons are reliable and practical to obtain. The WBT recorded consistently lower void content data than the DGT; it was also found to be less precise. For routine Carbon/Epoxy, void content determination, where relative comparisons are sufficient and high precision is not an issue, the WBT is recommended as it is practical to implement. When high precision is needed, the DGT is recommended. Image analysis methods produce highly localized data, but it is likely that they approximate true void content more closely than the DD/MD method because the void measurement, though actually a measure of void area, is direct. For more critical void content measurement where accuracy, as well as precision are required, a highly automated version of an image analysis technique, like the Omnimet, which scans a large number of cross sections is recommended. At present, this appears to be the best procedure available to determine true void content. DD/MD and image analysis approaches to void content determination are complimentary methods; the shortcomings of each are strengths of the other. Combining information from both methods is a superior means of characterizing voids in Carbon/Epoxy composites.